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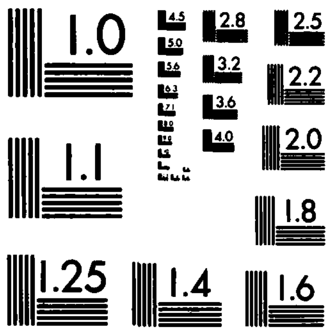

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PERMANENT MAGNET PROPERTIES OF IN SITU FORMED Cu-Fe MULTIFILAMENTARY COMPOSITES

By

G. Dublon, F. Habbal, and J.L. Bell

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Cu-Fe multifilamentary composites with up to 60 vol% Fe were prepared *in situ*. Magnetic hysteresis loops were obtained at room temperature as a function of composition, cross sectional area reduction, up to 99.9996%, and annealing conditions. H_{ci} and $(B \cdot H)_{max}$ increase with cross sectional area reduction and show pronounced changes on annealing. H_{ci} = 600, 520 and 380 Oe and M_r = 5.6, 8.2 and 11.9 kG were measured in the smallest 30, 45 and 60 vol% Fe composites, respectively, following optimal heat treatment. $(B \cdot H)_{max}$ = 3.2 MG·Oe was measured in both Cu-45 vol% Fe and Cu-60 vol% Fe with hysteresis loop squareness

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Permanent magnet properties of *in situ* formed Cu-Fe multifilamentary composites[a]

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ABSTRACT

Cu-Fe multifilamentary composites with up to 60 vol% Fe were prepared *in situ*. Magnetic hysteresis loops were obtained at room temperature as a function of composition, cross sectional area reduction, up to 99.9996%, and annealing conditions. H_{ci} and $(B \cdot H)_{max}$ increase with cross sectional area reduction and show pronounced changes on annealing. H_{ci} = 600, 520 and 380 Oe and M_r = 5.6, 8.2 and 11.9 kG were measured in the smallest 30, 45 and 60 vol% Fe composites, respectively, following optimal heat treatment. $(B \cdot H)_{max}$ = 3.2 MG·Oe was measured in both Cu-45 vol% Fe and Cu-60 vol% Fe with hysteresis loop squareness of 0.95. Considering the excellent mechanical and transport properties, inexpensive constituent elements and simple preparation, the *in situ* formed Cu-Fe composites appear to have the potential for permanent magnet applications.

PACS numbers: 85.70.Nk, 81.20.Jz, 75.60.Ej, 75,50.Bb

INTRODUCTION

Several methods have been developed during the last decades to prepare magnetically hard metal matrix composite materials containing aligned, submicron filaments [1-7]. Recently, intrinsic coercivities, H_{ci}, of up to ~ 600 Oe were measured at room temperature (RT) in *in situ* formed Cu-30 vol% Fe multifilamentary composites [7]. Combined with excellent mechanical [8] and transport properties [9,10] simple preparation [11] and inexpensive constituent elements, these materials appear to have the potential for permanent magnet (PM) applications. Further interest in the magnetic properties of the Cu-Fe *in situ* composites arises in conjunction with the extensive work on the isostructural superconducting Cu-Nb system [8-11]. The *in situ* formed composites with 10^6-10^{10} filaments/cm^2, each 50-2000 Å thick and up to several mm long, have an exceptionally high dislocation density, by far exceeding the maximum attainable densities in single-phase bulk materials [8-11].

This report is concerned mostly with the practical magnetic properties of the *in situ* formed Cu-Fe composite system. The understanding of the unusual magnetic, mechanical and transport properties, their

interdependence and relation to the unique microstructure of the in situ composite is, however, far from complete and requires extensive additional study.

In the present work we report RT permanent magnet (PM) characteristics of in situ formed Cu-Fe composites with 30, 45 and 60 vol% Fe.

EXPERIMENTAL

Multifilamentary Cu-Fe composites were formed in situ following a procedure which is described in detail elsewhere [8-11]. Starting two-phase alloys were prepared by RF levitation melting of 3N Cu and Fe and rapid cooling of the liquid solution. The resulting cylindrical ingots weighing ~ 12 g, ~ 1.2 cm in diameter, were then vacuum annealed for 12 days at 850°C to partially remove Fe from solid solution and in order to facilitate plastic deformation by cold swaging. The heat treatment produces significant coarsening of the initial microstructure as illustrated in Fig. 1 for Cu-60 vol% Fe. More work on the effects of annealing on the initial microstructure (and hardness) is needed as it in turn appears to affect the PM properties of the final, multifilamentary in situ composite.

Fig. 2 shows SEM micrographs of the cross section of Cu-30 vol% Fe and Cu-60 vol% Fe wire, 250 and 180 μ in diameter, respectively, as obtained by cold swaging and drawing with intermediate anneals at 300 or 350°C. Samples were eventually reduced down to 25 μ in diameter or 99.9996%. The estimated average filament cross section area in the smallest composites is $\sim 10^4$ Å^2. As expected for a bcc material in an fcc matrix, the initial Fe precipitates (Fig. 1) become ribbon-like filaments (Fig. 2) with a well defined <110> texture as they twist and curl due to the constraints of the surrounding matrix [8-11].

Hysteresis loops were obtained at RT using a vibrating sample magnetometer with the magnetic field applied parallel to the wire's long axis. The measurements were made as a function of composition, cold work and heat treatment (250-950°C).

RESULTS AND DISCUSSION

The RT saturation magnetization of the in situ formed Cu-Fe composite system increases linearly with the Fe content at 1.96 emu/g/vol% Fe between 5 and 60 vol% Fe and, within experimental accuracy, is unaffected by cold work or heat treatment beyond the first, high temperature anneal of the initial ingot. These results imply the presence of up to 2w%Fe dissolved in the Cu matrix following the first anneal - down from ~ 6 at % in the initial ingot - in agreement with

electron microprobe tests.

Figure 3 shows H_{ci} values of in situ formed Cu-45 vol% Fe composite wire at various stages of preparation. Similar results along with M_r/M_s values are shown in Fig. 4 for Cu-60 vol% Fe. Of particular interest is the dramatic increase of H_{ci} by more than a factor of 2 on proper annealing (Fig. 3). Such an increase of H_{ci} has already been reported for Cu-30 vol% Fe [7]. Similarly pronounced increases on annealing of the Young's modulus have been observed in isostructural in situ Cu-Nb composites [8]. Also, some of the increase of H_{ci} on annealing is lost by subsequent cold work (Fig. 3), resembling observed reversible changes of the elastic properties of Cu-Nb [8]. This, together with the stress dependence [10] of the superconducting properties of in situ formed Cu-Nb_3Sn and Cu-Nb_3Ga suggests the presence of stress induced magnetic anisotropy in Cu-Fe along with shape anisotropy. Work is in progress to determine the role of several conceivable sources of magnetic anisotropy [12] and the modes of magnetization rotation [13] in in situ formed Cu-Fe composites.

The pronounced increase, up to ~ 0.95, of both M_r/M_s (Fig. 4) and the hysteresis loop squareness with deformation and proper heat treatment implies improved filament alignment and uniformity. Anneals at high temperature (750-950°C) for only several minutes, or prolonged anneals at lower temperatures (300-500°C), produce a deterioration of the PM properties as a result of coarsening and eventual spheridization of the filaments. Some of that loss is recoverable by subsequent mechanical reduction. Additional indirect information about the composites' microgeometry is provided by the distribution of intrinsic coercivities as determined from their demagnetization remanence curves [14]. Thus obtained, the H_{ci} distributions in terms of filament volume fraction, v, for optimally annealed 30 and 60 vol% Fe samples are shown in Fig. 5. The analysis implies that ~ 80 vol% of filaments possess coercivities within 25 Oe of the measured composite H_{ci} in the best PM Cu-60 vol% Fe samples (Fig. 5). Similar results were obtained for Cu-45 vol% Fe. The smallest optimally annealed Cu-30 vol% Fe composites show a much broader distribution and some of the filaments appear to have coercivities up to ~ 1000 Oe (Fig. 5).

Figures 6 and 7 show RT hysteresis loops and energy product curves, respectively, of the best PM 30, 45 and 60 vol% Fe in situ composites prepared so far. The decrease of the best H_{ci} with increasing Fe content

is in agreement with general theoretical predictions [12]. However, in view of a maximum H_{ci} observed around 30 vol% Fe in powder metallurgically prepared Ag-Fe composites [2], there is work underway to determine the composition dependence of H_{ci} of <u>in situ</u> formed Cu-Fe down to 5 vol% Fe.

The PM properties of the Cu-Fe <u>in situ</u> composites (Figs. 6,7) are by far superior to those of powder metallurgically prepared composites [2-4]. In addition to their easier preparation, their PM properties are also better (Figs. 6,7) than those of conventionally prepared compacts [1]. Other structurally related materials, such as [15] Cu-1.7 w% Fe and [16] Fe-34 at % Pd precipitation alloys as well as Au-27 at % Co aligned eutectics [5] which exhibit higher coercivities [5,15,16] have, however, much lower remanence [5,15] and/or contain very expensive constituent elements [5,16]. Overall, taking into account coercivity, remanence, maximum energy product and hysteresis loop squareness, the PM characteristics of the Cu-Fe composites (Figs. 6,7) approach the performance of Cr-Co-Cu-Fe alloys [17] and sintered [18] Cr-Co-Fe. The Cu-60 vol% Fe material with H_{ci} up to 380 Oe, high remanence and square hysteresis loop (Fig. 6) compares favorably with commercial semi-hard magnets such as Remendur and Vicalloy as well as with more recently introduced [19] Co-Fe-Nb and [6] Fe-Ni and Fe-Mn alloys. Also, considering their exceptional mechanical strength [8], high electrical [9,10] and thermal [20] conductivity, superior to those of e.g. Co-Fe-V alloys [21], the <u>in situ</u> formed Cu-Fe composites appear to fulfill important requirements for rotor applications.

SUMMARY

<u>In situ</u> formed Cu-Fe multifilamentary composites with 30, 45 and 60 vol% Fe exhibit a range of useful permanent magnet properties. Intrinsic coercivities of 600, 520 and 380 Oe were measured at RT in optimally annealed 35-25 μ wire with 30, 45 and 60 vol% Fe, respectively. The 45 and 60 vol% Fe composites with remanences of 11.9 and 8.2 kG, respectively, hysteresis loop squareness of 0.95 and maximum energy product of 3.2 MG·Oe, compare favorably with Co-based and other semi-hard magnet alloys. In view of their outstanding mechanical and transport properties, in addition to inexpensive constituent elements and relatively simple preparation, the <u>in situ</u> formed Cu-Fe composites appear to have the potential for a variety of permanent magnet applications.

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FIGURE CAPTIONS

Fig. 1. SEM micrographs of polished and etched cross section of the initial Cu-60 vol% Fe ingot as cast (right) and annealed at 850°C for 12 days (left), showing the distribution of Fe precipitates (dark areas).

Fig. 2. SEM micrographs of the cross section of *in situ* formed Cu-60 vol% Fe 180 μ (right) and of Cu-30 vol% Fe 250 μ wire composite (left). The Fe filaments have been etched away (dark areas).

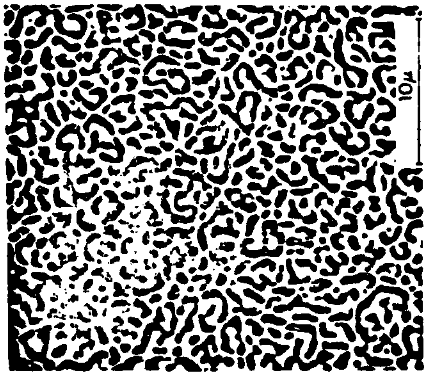
Fig. 3. Intrinsic coercivity of *in situ* Cu-45 vol% Fe wire composites as a function of $\eta = \ell n(a_o/a)$, where a_o and a are the cross section areas of the initial ingot and of the composite tested, respectively. (a), (b), (c), (d) denote H_{ci} values of samples obtained by successive wire drawing (full symbols) or anneals (open symbols). The effect of intermediate anneals is indicated by type of line drawn to connect data points for both as drawn and annealed samples.

Fig. 4. Intrinsic coercivity and remanence to saturation ratio of *in situ* Cu-60 vol% Fe wire composites as a function of $\eta = \ell n(a_o/a)$, where a_o and a are the cross section areas of the initial ingot and of the composite tested, respectively. Full lines indicate the succession of wire drawing and annealing steps taken, up to $\eta = 12$. Dashed and dotted lines connect H_{ci} data of samples annealed for 1 hour at 300 and 350°C, respectively.

Fig. 5. RT distribution of intrinsic coercivities in terms of filament volume fraction, v, as obtained from the demagnetization remanence curves of Cu-30 vol% Fe and Cu-60 vol% Fe *in situ* composites (see Ref. 14).

Fig. 6. RT hysteresis loops of optimally annealed Cu-30 vol% Fe 25 μ, Cu-45 vol% Fe 30 μ and Cu-60 vol% Fe 33 μ wire.

Fig. 7. RT energy product curves of the *in situ* Cu-Fe composites of Fig. 6.


10μ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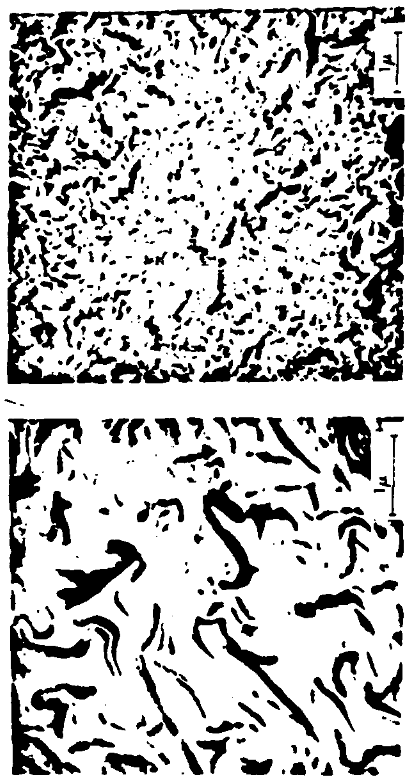

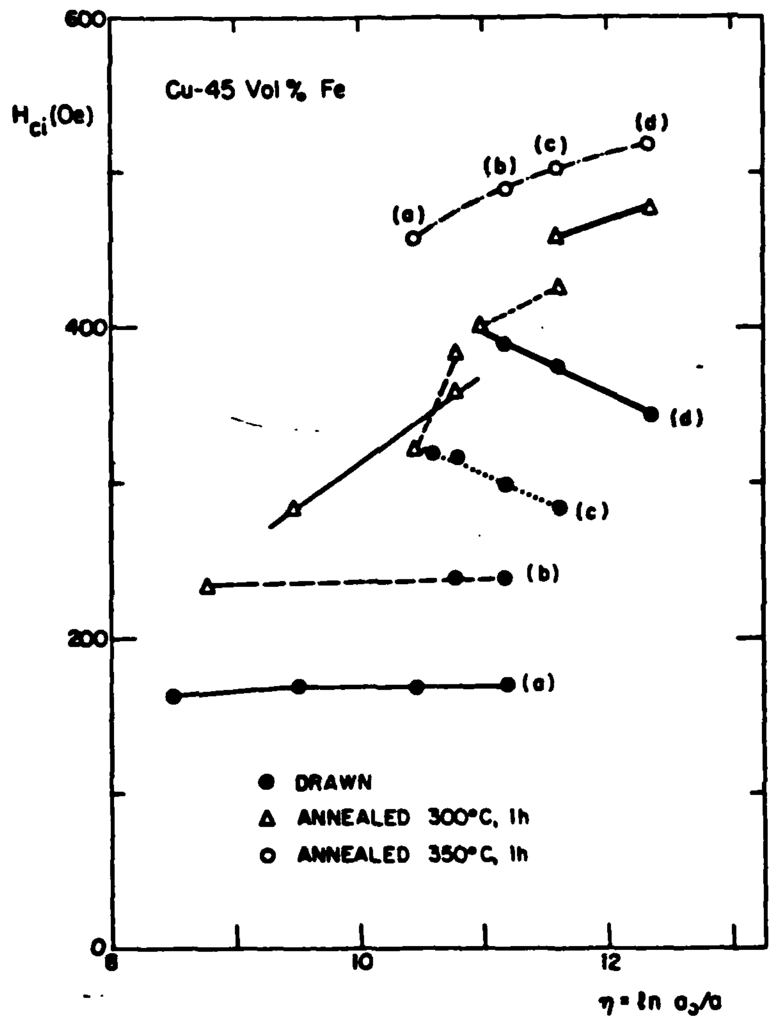
Cu-45 Vol % Fe
H_{ci}(Oe)
600
400
200
0
8
10
12
$\eta = \ln a_0/a$
(a)
(b)
(c)
(d)
DRAWN
ANNEALED 300°C, 1h
ANNEALED 350°C, 1h

F, 6, 2

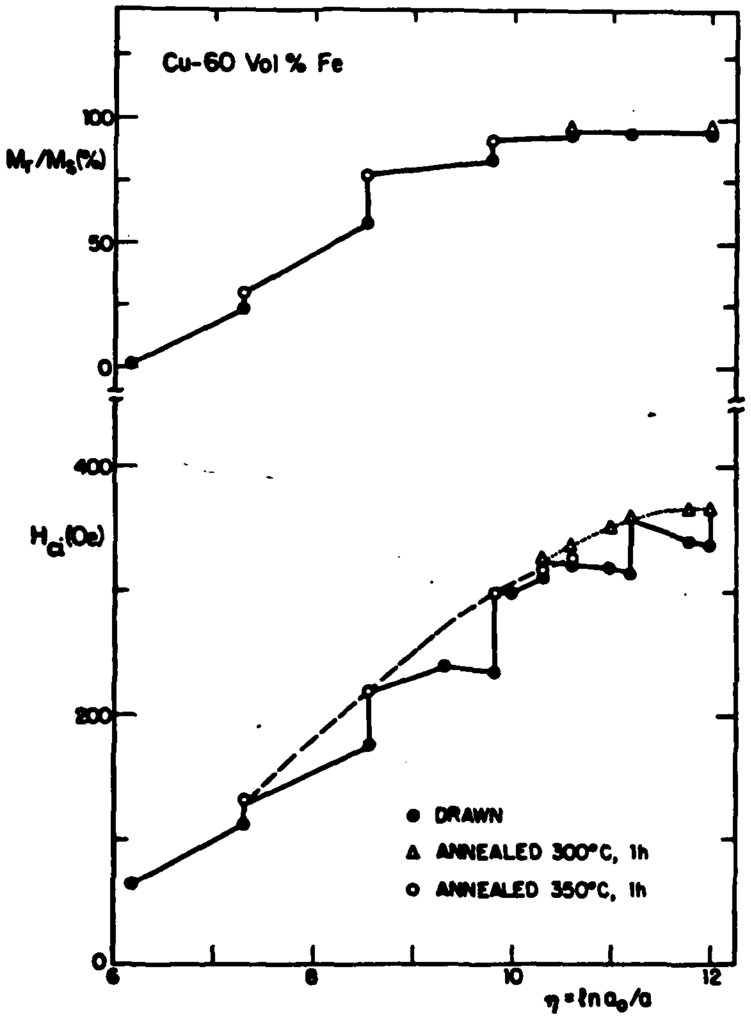
Cu-60 Vol % Fe
100
M_r/M_s(%)
50
0
400
H_{ci}(Oe)
200
0
6
8
10
12
$\eta = \ln a_0/a$
● DRAWN
△ ANNEALED 300°C, 1h
○ ANNEALED 350°C, 1h

Fig. 4

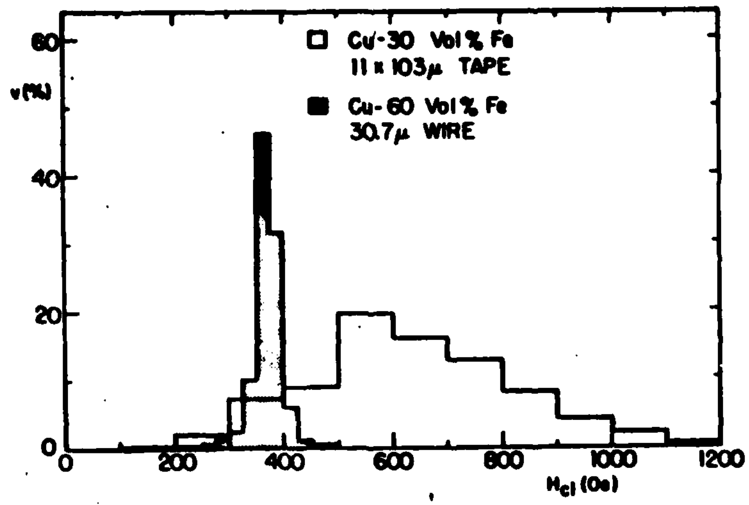
Cu-30 Vol % Fe
11 x 103μ TAPE
Cu-60 Vol % Fe
30.7μ WIRE
v(%)
60
40
20
0
0
200
400
600
800
1000
1200
Hcl (Oe)

Fig. 5

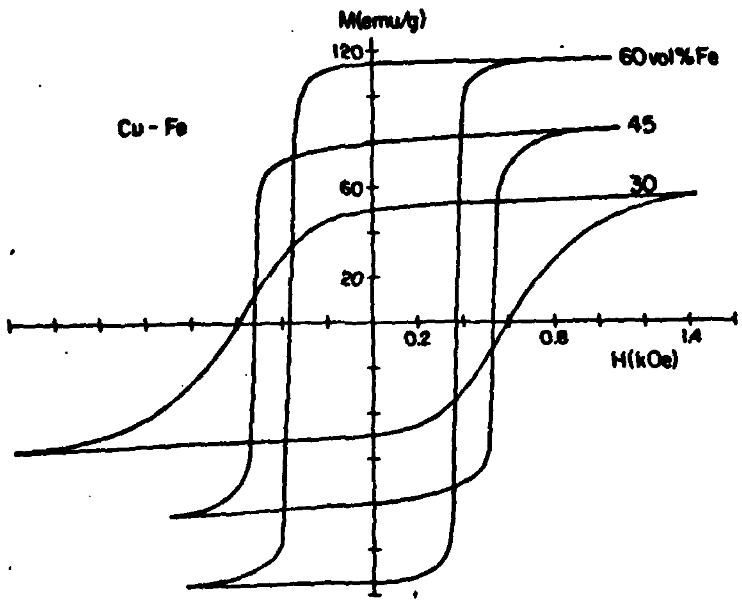

Fig. 6

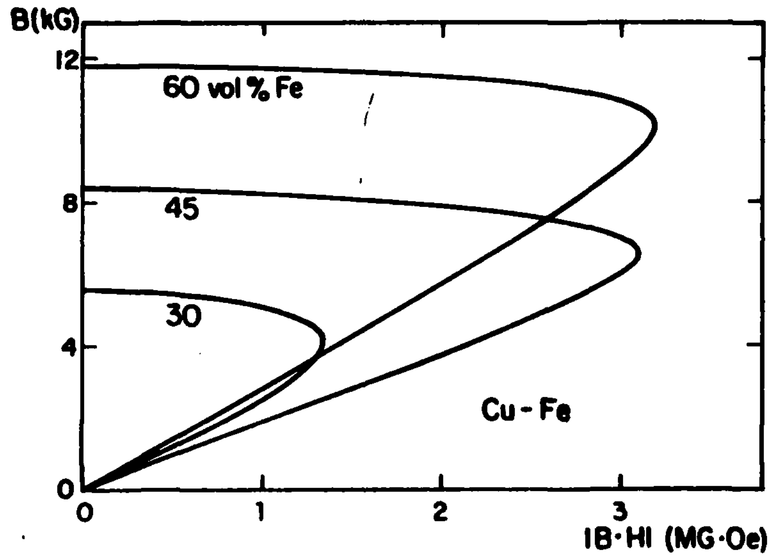

Fig. 7

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